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(54) Title: FLAVOUR ENCAPSULATION

(57) Abstract: A process for the preparation of an encapsulated flavour composition comprising a flavour encapsulated in a high-boiled sugar glass which comprises boiling the ingredients of a high-boiled sweet to give a syrup, heating the syrup to reduce the moisture content to below 5 %, cooling the syrup to a temperature from 120- 80 °C, adding the flavour with agitation to encapsulate the flavour, cooling to solidify the flavour, and finally particulated.

FLAVOUR ENCAPSULATION

FIELD OF THE INVENTION

- 5 The present invention relates to the encapsulation of flavours, more especially to the encapsulation of volatile flavours by a high-boiled sugar glass, and to edible products containing such encapsulated flavours.

BACKGROUND OF THE INVENTION

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US-A-5,009,900 describes a process for encapsulation of a composition containing at least one volatile and/or labile component, which process comprises forming a mixture of said composition with:

- 15 (a) from about 40 to 80% by weight of a water-soluble, chemically-modified starch having a dextrose equivalent not greater than about 2;
(b) from about 10 to 40% by weight of a maltodextrin having a dextrose equivalent in the range of from about 5 to about 15;
(c) from about 5 to about 20% by weight of corn syrup solids or a polydextrose having a dextrose equivalent in the range of from about 21 to about 42; and
20 (d) from about 5 to about 20% by weight of a mono- or disaccharide, all of said percentages being based upon the total weight of said components (a), (b), (c) and (d);
and extruding the mixture to form a glassy matrix having a glass transition temperature of at least 40°C wherein the composition containing at least one
25 volatile and/or labile component is encapsulated.

WO 96/11589 describes a process for the preparation of particulate flavour compositions comprising a flavour oil fixed in a particulate polyol material, which comprises:

- 30 (a) mixing a mono- or disaccharide, a polysaccharide and water with a minor but flavour effective amount of a flavour oil to form a homogeneous substrate such that the resulting particulate flavour compositions possess a T_g below room temperature; and
(b) extruding said homogeneous substrate at a temperature sufficient to form a
35 melt which on cooling solidifies as a hard rubbery and amorphous material having said flavour oil entrapped therein

The above processes, as are many of the prior known processes for encapsulating flavours, require extrusion. In many cases, this requires investment in costly and space-consuming extrusion equipment as well as the know-how to operate the
5 extrusion system.

High boiled sugar glass is predominantly an amorphous glassy matrix at ambient temperature with the sugar in a continuous phase. In contrast, pressed or compressed sweets and tablets, etc. contain sugar particles which are not
10 continuous and any flavour contained therein can be lost easily. We have found, surprisingly, that by using high-boiled sugar glass (e.g. Fox's glacier mint) to encapsulate volatile flavour for use as a flavour protector/carrier, e.g. in edible products such as pressed sweets or tablets, we can use a conventional
15 manufacturing process for the production of high-boiled sweets to produce the sugar glass without the need of expensive extruders. We have found surprisingly that not only does the sugar glass restrict the mobility of flavour molecules at ambient temperatures so that flavour losses are minimised during mixing but also, the flavours are retained during shelf storage.

20 SUMMARY OF THE INVENTION

According to the present invention there is provided a process for the preparation of an encapsulated flavour composition comprising a flavour encapsulated in a high-boiled sugar glass which comprises boiling the ingredients of a high-boiled
25 sweet to give a syrup, further heating the syrup to reduce the moisture content to below 5% by weight based on the weight of the syrup, cooling the syrup to a temperature from 120° to 80°C, adding the flavour with agitation to encapsulate the flavour, cooling to solidify the encapsulated flavour, and finally particulating the encapsulated flavour.

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DETAILED DESCRIPTION OF THE INVENTION

The ingredients of the high-boiled sweet and the amounts in which they are used are well known to the skilled person and generally comprise a mixture of a sugary
35 or non-sugary sweet material with water, e.g. from 0.1-2.5% by weight of water.

Examples of sugary sweet materials are sucrose and corn/glucose syrup.

Examples of non-sugary sweet materials include isomalt, lactitol, maltitol and lycasin.

5

after boiling the ingredients of the high-boiled sweet to give a syrup, the syrup may be further heated to a temperature from 120°C to 160°C to reduce the moisture content, preferably to from 0.1 to 4.5% and more preferably to from 1 to 3% by weight. the actual temperature may depend on the type of cooker used. for
10 example, if an open pan is used, the syrup may conveniently be heated to from 145° to 160°C. if a vacuum cooker is used, the syrup may conveniently be heated to a temperature from 125° to 140°C depending on the vacuum. if a batch cooker is used, the syrup may conveniently be heated to a temperature from 135° to 150°C.

15 After the water content has been reduced to below 5% , the syrup is preferably cooled to from 110° to 82.5°C, more preferably from 105° to 85°C, and especially from 100° to 87.5°C. The flavour is then added to the syrup with mixing and the syrup cooled further preferably to room temperature, e.g. from 20° to 30°C, to solidify the high-boiled sugar glass containing the encapsulated flavour, e.g. by
20 pouring onto a water-cooled slab.

The flavour is advantageously a volatile flavour which may be in liquid or solid form, e.g. vanilla, orange, chocolate, coffee, cocoa, and especially a flavour oil, e.g. mint oil, orange oil, cinnamon, hazelnut, etc. If desired, a colour or an acid
25 (such as in Foxes fruits (citric and lactic acids) may be incorporated in addition to the flavour. The colour can be any format and may be water soluble, e.g. dyes, pigments or it may be a non soluble fat-based or oil which would be added as a dispersion. The acid may be, for instance, citric, malic, tartaric, lactic or ascorbic acid. The amount of colour will depend on the legal maximum levels permitted
30 and may be any amount up to the maximum for that particular colour or blend of colours. The amount of acid may be from, for example, 0.1-3%. The colour or acid may be added to the sugar glass at the same time as the flavour or separately anytime after heating to 120°C. The acid, with or without the addition of buffer, should be a food-acceptable acid such as the acid used in Fox's fruits.

35

The amount of the flavour added to the cooled syrup should be an amount effective to impart the desired flavour to the edible product in which the encapsulated flavour composition is incorporated. The amount of flavour added may be up to 20%, e.g. from 0.1 to 20%, preferably from 0.5 to 17.5% and more preferably from 2 to 15% by weight based on the weight of the encapsulated flavour composition.

After the flavour has been added and mixed with the cooled syrup to encapsulate it, the encapsulated mix is cooled to ambient temperature and particulated into beads, slabs or a powder. The particle sizes of the beads, slabs and powders depend on the application, e.g. to incorporate into a pressed sweet, this may be less than 1mm. In some cases, they will be added to give a visual impact for the colour and appearance. For visual effect the particle size may be in the range of from 50 microns to 5mm depending on the effect or texture required. to use as a concentrated flavour sugar for beverages, the size will be of any size depending on the dissolving characteristics., Particulation into beads may be carried out by spray chilling/drying. Slabs may be formed by injection moulding or depositing into a mould. Powders may conveniently be obtained by grinding or with addition of a spray drying process.

20

The present invention also provides an encapsulated flavour composition comprising a flavour encapsulated in a high-boiled sugar glass.

The encapsulated flavour composition may be used in any desired application in edible products.

25

Accordingly, the present invention also provides an edible product having incorporated therein or covered therewith an encapsulated flavour composition comprising an effective amount of a flavour encapsulated in a high-boiled sugar glass. Preferably, the encapsulated flavour composition is prepared by the process of the present invention.

30

The amount of encapsulated flavour composition may be from 0.1% to 10% and preferably from 1 to 5% by weight based on the weight of the edible product.

35

The edible products include confectionery, pharmaceutical, beverage or functional products. Confectionery products may include pressed tablets or sweets, chew and hydrocolloid based products. Pressed tablets or sweets conventionally contain mainly granulated sugar together with a binder, a lubricant and a optionally a colour/conventional flavour. Examples of binders are gums such as gum arabic, gelatin or alginates, or they may be starches, sugars or fats. Examples of lubricants are magnesium stearate, stearic acid, oils or fats.

When the encapsulated flavour composition is used to cover the edible product, it can be used to dust, coat or surface enrobe the edible product. In this case, the edible product is preferably a confectionery product.

When used in beverages, the encapsulated flavour composition will allow instant release of flavour when dissolving in water before consumption and the present invention also provides a beverage containing an effective amount of an encapsulated flavour composition comprising a flavour encapsulated in a high-boiled sugar glass.

The encapsulated flavour composition may be also contain a colour or and acid and used as a colour/flavour/acid encapsulated compound in edible products.

The encapsulated flavour composition can encapsulate a flavour to prevent deterioration of flavour intensity and characteristics over time in foods and pharmaceutical products.

High boiled sugar glass being predominantly an amorphous glassy matrix at ambient temperature restricts the mobility of flavour molecules. Hence, this may be able to protect the product from flavour loss leading to maximum flavour retention especially for volatile flavour (e.g. essential oil). This may be especially useful for products which are stored at high humidity and temperature. e.g. in tropical regions or South American climates.

The issue of hygroscopicity could be overcome by adding a small amount, e.g. from 0.1 to 5% by weight of a biopolymer e.g. maltodextrin to increase the glass transition temperature.

The depositing conditions may require modification to suit the higher viscosity as a consequence of addition of the biopolymer into a mould whereby "tailing" should be avoided after the flavour addition.

5 The present invention provides the following advantages:

- 1) It improves the product quality,
- 2) It increases the fresh and shelf life of the products,
- 3) It reduces waste and increases efficiency due to possible use of rework from
10 existing high boiled products,
- 4) It enables utilization of existing assets, using current high boil process and equipment for production of flavour compounds for foods and pharmaceutical applications,
- 5) It reduces cost for flavour as losses are minimised.
- 15 6) It enhances visual appear and perceived quality.

EXAMPLES

20 The following Examples further illustrate the present invention. Percentages are given by weight.

Example 1

25 The recipe used for a high boiled sugar glass is as follows:

	%
Sugar	56
42DE Glucose syrup	24
Water	20

30

The above ingredients were boiled and further heated to 149°C in a vacuum cooker to reduce the moisture content to 2.5% and allowed to cool to 90°C. 5% mint oil was then added with mixing, and the syrup cooled further to room temperature to solidify the high-boiled sugar glass containing the encapsulated mint
35 flavour by pouring onto a cold water-cooled slab. The sugar glass was broken and

ground in a Stephan Mixer to a fine powder having an average particle size of 0.1 to 1mm

Example 2

5

A basic recipe for sugar-free high boiled sugar is as follows:

	%
Isomalt	80
Water	20

10

The above ingredients were boiled and further heated to 155-160°C in a vacuum cooker to reduce the moisture content to 1.5% and allowed to cool to 90°C. 5% mint oil was added with mixing, and the syrup cooled further to room temperature to solidify the high-boiled sugar glass containing the encapsulated by pouring onto a cold water-cooled slab. The sugar glass was broken and ground in a Stephan Mixer to a fine powder having an average particle size of 0.1 to 1mm.

15

Example 3

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5 parts of the particulated sugar glass containing encapsulated mint flavour prepared as in Example 1 is added with mixing to 95 parts of the ingredients of a conventional pressed tablet or sweet containing mainly granulated sugar together with a binder, a lubricant and a optionally a colour/conventional flavour. The mixture is then compressed under pressure in a die to give a mint flavoured pressed sweet.

25

CLAIMS.

1. A process for the preparation of an encapsulated flavour composition comprising a flavour encapsulated in a high-boiled sugar glass which
5 comprises boiling the ingredients of a high-boiled sweet to give a syrup, heating the syrup to reduce the moisture content to below 5%, cooling the syrup to a temperature from 120 to 80°C, adding the flavour with agitation to encapsulate the flavour, further cooling to solidify the encapsulated flavour, and finally particulating the encapsulated flavour.
- 10 2. A process according to claim 1 wherein the ingredients of the high-boiled sweet comprise a mixture of a sugary or non-sugary sweet material with water.
3. A process according to claim 2 wherein the sugary sweet material is sucrose or corn/glucose syrup.
- 15 4. A process according to claim 2 wherein the non-sugary sweet material is isomalt, lactitol, maltitol or lycasin.
5. A process according to claim 1 wherein after boiling the ingredients of the high-boiled sweet to give a syrup, the syrup is heated to a temperature from 120°C to 160°C to reduce the moisture content.
- 20 6. A process according to claim 1 wherein after the water content has been reduced to below 5%, the syrup is cooled to from 120° to 80°C.
7. A process according to claim 1 wherein the flavour is a volatile flavour which is in liquid or solid form.
8. A process according to claim 1 wherein the flavour is vanilla, orange, chocolate, coffee, cocoa, mint oil, orange oil, cinnamon or hazelnut.
- 25 9. A process according to claim 1 wherein a colour or an acid may be incorporated in addition to the flavour
10. A process according to claim 1 wherein the amount of the flavour added to the cooled syrup is up to 20% by weight based on the weight of the encapsulated flavour composition.
- 30 11. A process according to claim 1 wherein after the flavour has been added and mixed with the cooled syrup to encapsulate it, the encapsulated mix is cooled to ambient temperature and particulated into beads, slabs or a powder.
12. An encapsulated flavour composition comprising a flavour encapsulated in a high-boiled sugar glass.

13. An edible product having incorporated therein an encapsulated flavour composition comprising an effective amount of a flavour encapsulated in a high-boiled sugar glass.
14. An edible product according to claim 13 wherein the edible product includes confectionery, pharmaceutical, beverage or functional products.
15. An edible product according to claim 13 wherein confectionery products include pressed tablets, chew and hydrocolloid based products.
16. An edible product which is dusted, coated or surface-enrobed by an encapsulated flavour composition comprising a flavour encapsulated in a high-boiled sugar glass.
17. A beverage containing an effective amount of an encapsulated flavour composition comprising a flavour encapsulated in a high-boiled sugar glass.
18. An encapsulated flavour composition according to claim 12 comprising from 0.1 to 5% by weight of a biopolymer.

INTERNATIONAL SEARCH REPORT

In: ational Application No

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A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A23L1/22 A23G3/00 A23G3/02 A23L2/56

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A23L A23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, FSTA

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 794 741 A (WEIGLE D) 26 February 1974 (1974-02-26)	1,5-13, 18
Y	the whole document	2-4
X	EP 0 076 115 A (WARNER LAMBERT CO) 6 April 1983 (1983-04-06) page 5, line 2 -page 7, line 24; claims; examples 3,7	1,5-15
X	EP 0 370 296 A (PROCTER & GAMBLE) 30 May 1990 (1990-05-30)	13-15
Y	page 3, line 27 - line 39 page 3, line 44 - line 50 page 6, line 5 - line 14; claim 1	2-4
Y	US 4 134 999 A (MUHLER JOSEPH C ET AL) 16 January 1979 (1979-01-16) column 2, line 29 -column 3, line 56	2-4
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>US 3 615 645 A (FORKNER JOHN H) 26 October 1971 (1971-10-26) -----</p>	

INTERNATIONAL SEARCH REPORT

Information on patent family members

In ational Application No

PCT/EP 01/02265

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 3794741	A	26-02-1974	CA 1000998 A	07-12-1976
EP 0076115	A	06-04-1983	AU 8869482 A	31-03-1983
			ES 515940 D	01-01-1984
			ES 8501615 A	01-03-1985
			JP 58076062 A	09-05-1983
EP 0370296	A	30-05-1990	AT 100280 T	15-02-1994
			AU 638951 B	15-07-1993
			AU 4575989 A	31-05-1990
			CA 2003188 A,C	25-05-1990
			CN 1043070 A,B	20-06-1990
			DE 68912529 D	03-03-1994
			DE 68912529 T	26-05-1994
			DK 590689 A	26-05-1990
			ES 2061888 T	16-12-1994
			FI 95532 B	15-11-1995
			HK 1006133 A	12-02-1999
			IE 62192 B	28-12-1994
			IN 172980 A	15-01-1994
			JP 2227034 A	10-09-1990
			JP 2859331 B	17-02-1999
			KR 141920 B	01-06-1998
			MX 172577 B	03-01-1994
			NO 176691 B	06-02-1995
			NZ 231513 A	25-11-1992
			PH 26470 A	27-07-1992
			PT 92387 A,B	31-05-1990
			US 5017385 A	21-05-1991
US 4134999	A	16-01-1979	NONE	
US 3615645	A	26-10-1971	NONE	

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